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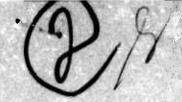
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TECHNICAL REPORT AFATL-TR-75-17

EVALUATION OF RHEOLOGICAL PROPERTIES

OF

FLAME FUELS

USING A CAPILLARY EXTRUSION RHEOMETER

FAE AND FLAME BRANCH MUNITIONS DIVISION

JANUARY 1975

FINAL REPORT: JANUARY 1973 TO JULY 1974

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18. SUPPLEMENTARY NOTES

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

O. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report describes a research effort to develop methods to study the flow properties, such as viscosity and elasticity, of polymer solutions used as flame agents. Viscosity of the polymer formulations were studied at -3.89°C (ASCP) using a capillary extrusion rheometer. Several brands of gasoline were used to prepare the polymer solutions, which gave varied flow curves under increased shear stresses. The flow curves exhibited considerable variation when different brands of gasoline were used to prepare

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the polymer solutions. The capillary length and die swell methods were used to study the elastic properties of the polymer solutions.



PREFACE

This technical report is based on a study conducted at the Air Force Armament Laboratory, Armament Development and Test Center, in support of Project 10820302 during the period from January 1973 to July 1974.

This technical report has been reviewed and is approved for publication.

FOR THE COMMANDER

WILLIAM F. BROCKMAN, Colonel, USAF

Chief, Munitions Division

TABLE OF CONTENTS

Section	Page
I	INTRODUCTION 5
II	THEORETICAL
	Shear Stress
	Shear Rate
	Viscosity
	Elasticity
111	EXPERIMENTAL
	Apparatus
	Techniques
	Materials
	Data Reduction
IV	RESULTS AND DISCUSSION
	Calibration of Equipment
	Comparison of Recoverable Shear and Memory 23
	Rheological Behavior of Napalm B 23
	Rheological Behavior of Experimental
	Formulation
v	SUMMARY AND CONCLUSIONS
Appendix A	CAPILLARY EXTRUSION RHEOLOGY DATA REDUCTION
	PROGRAM 1939
	SYMBOLS
	REFERENCES

LIST OF FIGURES

Figure	Title	Page
1	Log Shear Rate versus Log Shear Stress of Various Capillaries at 23.9°C	11
2	Pressure (Dynes) versus L/D of Two Different Size Capillaries at Four Different Shear Rates at 23.9° C	12
3	Recoverable Shear versus Shear Rate for Napalm B	1.3
4	Percent Memory versus Shear Rate for Napalm B	14
5	Capillary Extrusion Rheometer	17
5	Extrusion Rams used in the Study	17
7	Capillaries used for the Recoverable Shear (S_r) Study	18
8	H/P 5325B Electrical Counter	20
9	Modified Syringe with Tubing	21
10	Flow Curves of Various Capillaries for Napalm B	24
11	Flow Curve for Napalm B with Chevron Gasoline at OC (32°F)	25
12	Comparison of Percent Memory and $S_{\mathbf{r}}$ for Napalm B	26
13	Flow Curve for a 46 Percent Styrene-Methylmethacrylate at 23.9°C	27
14	Percent Memory of Napalm B (Now Mixed)	29
15	Flow Curve of Napalm B Formulated with Various Gasolines.	30
16	Percent Memory of Various Gasolines and Polystyrene Formulations	31
17	Flow Curve of Napalm B and Styrene-Methylmethacrylate at 25°F and 75°F	32
18	Comparison of Percent Memory for Napalm B and a 40 Percent Formulation of Styrene-Methylmethacrylate (NAS).	33
19	Comparison of Percent Memory for Napalm B and 29 Percent SBR 40 Solution	35

LIST OF FIGURES (CONCLUDED)

Figure	Title		
20	Comparison of Viscosity versus Shear Rate of 29 Percent SBR 40	36	
21	Comparison of Viscosity for Napalm B and a 29 Percent SBR 43 Formulation	37	
22	Comparison of Percent Memory for Napalm B and 29 Percent SBR 43 Formulation	38	

SECTION I

INTRODUCT ION

Requirements for firebombs include a capability for high speed delivery over a wide temperature range. High speed delivery conditions expose the firebomb fuel to high wind shear forces as well as the explosive forces used in dissemination of the fuel. In addition, temperature dependence of the flow behavior of a thermoplastic material such as the polystyrene in Napalm B results in large variations in its properties.

The rheological properties of concentrated polymer solutions are of major importance in the development of improved flame agents for these high speed delivery conditions over a wide temperature range. An understanding of the rheological behavior of standard agents (such as Napalm B) and candidate systems will enable researchers to predict the behavior of candidate fuels based on laboratory studies, as well as providing guidance as to the type of materials which should be investigated.

The rheological properties which are most important to fuel behavior are the viscosity and elasticity (termed recoverable shear or percent memory) as a function of shear rate. Gaskins has correlated shear rate with the velocity of a gelled hydrocarbon extruded at high speeds from a nozzle. Based on these calculations, it has been estimated that exposure of a fuel to an air stream at 900 feet per second is equivalent to a shear rate in the range of $10^6 \ {\rm sec}^{-1}$.

Numerous researchers have studied the viscosity of flame agents with rotational viscometers such as the Brookfield or MacMichael instruments; however, these instruments are limited to low shear rate ranges and require exposure or the sample to the atmosphere during the measurement. Gaskins has designed a capillary extrusion rheometer to study both viscosity and recoverable shear as a function of shear rate. However, the instrument utilized is limited in shear rate to about $10^5 \ {\rm sec}^{-1}$ and requires numerous runs to overcome data scatter. The technique utilized for studying recoverable shear involves a large number of laboratory experiments and a long data reduction process.

Reference

^{1.} Gaskins, Frederick. A Study of Jet-Tension Instrument and Mechanical Flame Thrower. Franklin Institute Report F-A 1978.

^{2.} Gaskins, Frederick. Rheological Properties and Performance of Napalm B in Comparison to Standard Flame and Incendiary Agents (U). EATR 4155, February 1968.

The objective of this study was to develop simple instrumentation and reproducible techniques necessary to study the rheological properties of polymer solutions. A parallel, contractual effort ³, ⁴ was utilized in part to develop the required instrumentation.

To establish the procedures and provide a data baseline for comparison with experimental formulations, an extensive investigation of Napalm B has been included in this study.

Reference

^{3.} Long, R. L. <u>Flame Agents for High Velocity/Low Temperature Use (U)</u>. Air Force Armament Laboratory Report AFATL-TR-71-55. Monsanto Research Corporation, May 1971.

^{4.} Long, R. L. <u>Improved Flame Agents (U)</u>. Air Force Armament Laboratory Report AFATL-TR-72-177. Monsanto Research Corporation, September 1972.

SECTION II

THEORETICAL

Rheology is the science of the deformation and flow of matter. Comprehensive theoretical discussions of rheology are adequately discussed in the literature. A limited explanation of the parameters studied and the equations used to calculate these parameters will be included.

The deformation of a body can be arbitrarily divided into two general types: (1) spontaneously reversible deformation called elasticity, and (2) irreversible deformation called flow or viscous behavior. In an idealized case, if deformation is carred out infinitely slow, there will be no viscous contribution and only elastic effects will show up. On the other hand, in continuous, steady-state flow at a uniform rate there will be no elastic contribution and the entire effect will be viscous.

The polymer solutions studied in this program exhibit both reversible deformation or elasticity and irreversible deformation or flow and are called viscoelastic materials.

SHEAR STRESS

To produce flow or an elastic strain, a stress, defined as a force per unit area, must be applied. In a capillary extrusion rheometer, it is assumed that steady flow is obtained in the capillary and that all the forces applied to the solution cause flow. In this case, the viscous forces tending to retard the flow will be exactly balanced by the force resulting from the pressure differential between the two ends of the capillary. The shear stress in a fluid flowing through a capillary is directly proportional to the distance from the center of the capillary, varying from zero shear stress at the center to a maximum at the wall. The most convenient location for measurement of the shear stress is at the wall of the capillary. The shear stress applied at the wall in the capillary extrusion rheometer used in this study is calculated from the following equation:

$$T = \frac{Fr}{2 R^2 L}$$

Reference

^{5.} Van Wazer, J. R., et. al. <u>Viscosity and Flow Measurement</u>. Interscience Publishers, 1963.

where: $T = Shear stress, dynes/cm^2$

F = Force applied (ram load, dynes)

r = Orifice radius, cm

R = Barrel radius, cm

L = Orifice length, cm

SHEAR RATE

The rate of deformation for flow is a function of shear. Simple shear can be considered as a process in which infinitely thin, parallel plates slide over each other.

Viscous deformation is expressed in terms of rate of shear, which is the change in velocity of flow with a distance measured at right angles to the direction of flow. In the capillary rheometer, the rate of shear also varies with the radius of the capillary and must be calculated at the same point as the shear stress for the construction of flow curves. The shear rate at the wall is a more difficult quantity to determine from experimental data than shear stress. For the calculation of shear rate at the wall in a capillary extrusion rheometer, the Rabinowitsch⁶ correction factor is applied to the rate of shear which has been calculated from the volumetric flow rate of the fluid, according to the following equation:

$$\dot{\gamma}_{\dot{\omega}} = \left(\frac{4Q}{\pi r 3}\right) \left(\frac{3+b}{4}\right)$$

where: $\dot{\gamma}_{\omega}$ = Corrected shear rate, \sec^{-1}

Q = Extrusion rate in cm³/sec⁻¹ = volume extruded/ extrusion time

r = Orifice radius, cm

 $\left(\frac{3+b}{4}\right)$ = Rabinowitsch correction factor, where b is the slope of the flow curve (log shear rate versus shear stress)

Reference

6. Rabinowitsch, B., Z. Physik, Chem., A 145, 1 (1929).

The ratio of applied shearing stress to rate of shear for ideal viscous bodies is called the coefficient of viscosity or, more commonly, viscosity. This viscosity is a measure of the resistance to flow and can be expressed as:

$$Viscosity = \frac{Shear \ stress}{Shear \ rate}$$

or

$$N = \frac{T}{\dot{Y}}$$

The ideal viscous body is the Newtonian fluid for which the coefficient of viscosity is a constant. If the viscosity changes with shear rate or shear stress, it is called non-Newtonian or apparent viscosity. Most solutions of polymers, such as polystyrene, exhibit an apparent viscosity that is constant at low shear rates and then decreases in magnitude as the shear rate is increased. This decrease in viscosity as the shear rate is increased is called shear thinning or pseudoplastic flow.

ELASTICITY

The calculation of shear stress, shear rate, and viscosity from the equations shown assumes that the energy input to the solution, or the pressure drop across the capillary, is primarily utilized to overcome viscous resistance to flow, and that all other effects are small and can be neglected. Since the capillary has rigid walls, the elastic deformation of the fluid is minimized. However, when the fluid emerges from the capillary, it is no longer restrained by the walls, and energy which was reversibly stored is recovered by expansion of the fluid. Thus, highly viscoelastic materials may have elastic components that reversibly absorb a portion of the energy input. Philippoff and Gaskins have discussed this elastic energy correction and have developed a graphical procedure for the calculation of recoverable shear stress, based on the capillary length.

a. Recoverable Shear

In order to determine the elasticity or recoverable shear by the capillary length technique, the polymer solutions are extruded through capillary tubes of the same diameter but of different lengths. Flow curves (log shear rate versus log shear stress) are then prepared for each capillary. From these plots, the pressure differential (ΔP) required

Reference

7. Philippoff, W. and Gaskins, F. H., "The Capillary Experiment in Rheology," Transactions of the Society of Rheology, II (1958), p. 263-284.

for each capillary with a different length and radius (L/r) ratio is determined at various levels of shear rate. Figure 1 is an example of this type plot for Napalm B at 23.9°C (75°F). This procedure is required to obtain the data for each capillary over a variation of shear rates. Plots are then made of ΔP versus the L/r ratio for each selected shear rate, as in Figure 2. For each of the shear rates plotted, there is an initial pressure drop (ΔP) at L/D = 0 which is a combination of the entrance losses and the pressure input that is reversibly stored and can be recovered outside the capillary. Flow curves of data collected with various capillaries give a curve independent of capillary dimensions, indicating that the end effects are negligible; thus, the initial pressure drop is primarily the pressure input that is reversibly stored and can be recovered outside the capillary. Philippoff and Gaskins have shown that the most direct way of determining the elastic energy function is by measuring the x-intercept (as in Figure 2) and calculating the recoverable shear as:

$$S_r = -2 (x-intercept)$$

The recoverable snear is then plotted against shear rate as in Figure 3.

b. Memory

Monsanto Research Corporation⁵ has shown that the elasticity of a polymer solution can also be studied by measuring the expansion of the solution as it is extruded from the capillary. The basic technique for determining the die swell or memory of polymer melts is the measuring of the diameter of the extruded polymer strand. For the solutions studied in this program, the liquid strand is photographed as it emerges from the capillary and the measurements are made from the photograph. The diameter of the extruded strand, D_1 , and the capillary orifice diameter, D_0 , are used to calculate the percent memory by the following equation:

Percent memory =
$$\frac{D_1 - D_0}{D_0}$$
 X 100

From the equation it can be seen that if the extruded strand is the same diameter as the orifice, the solution has no recoverable shear and the percent memory will be zero. The percent memory is plotted as a function of the shear rate as shown in Figure 4 for Napalm B at 23.9°C (75°F).

c. Comparison of Recoverable Shear and Memory

The elastic nature of a polymer solution can be studied by either the capillary length or memory technique. In the capillary length technique, the energy which is recoverably stored in the solution is measured. While the theoretical basis of the technique is sound, a considerable number of

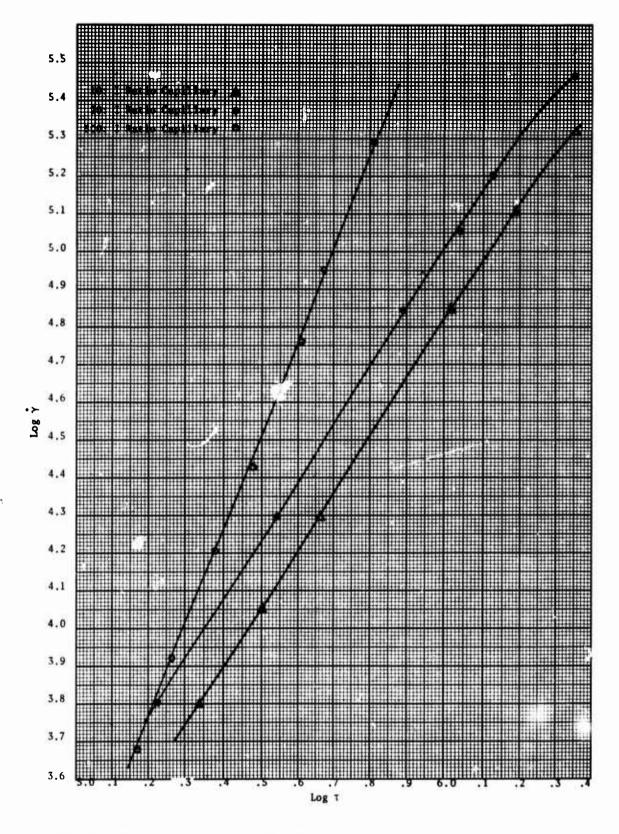
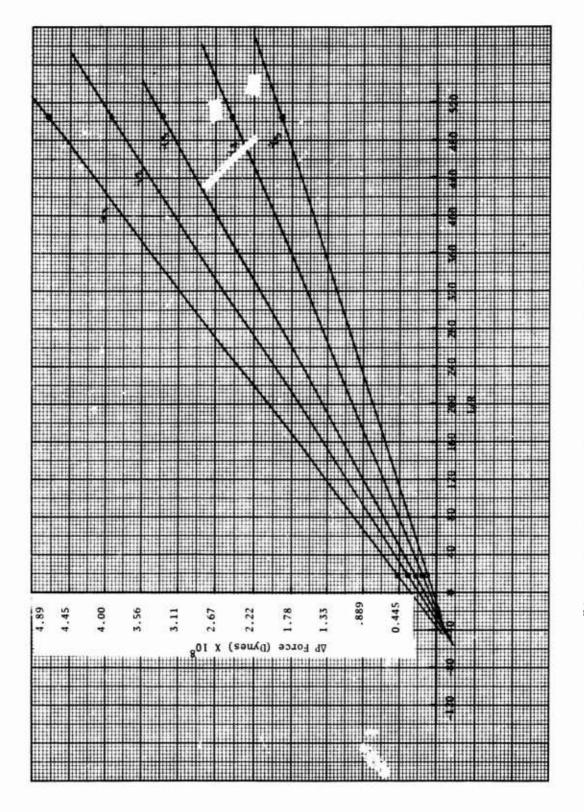


Figure 1. Log Shear Rate versus Log Shear Stress of Various Capillaries at 23.9°C



Pressure (Dynes) versus L/D of Two Different Size Capillaries at Four Different Shear Rates at 23.9°C 5. Figure

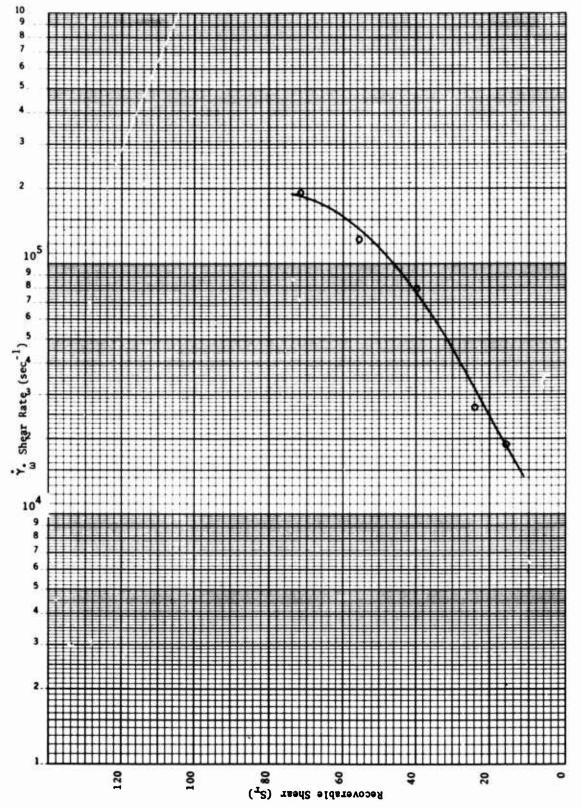


Figure 3. Recoverable Shear versus Shear Rate for Napalm B

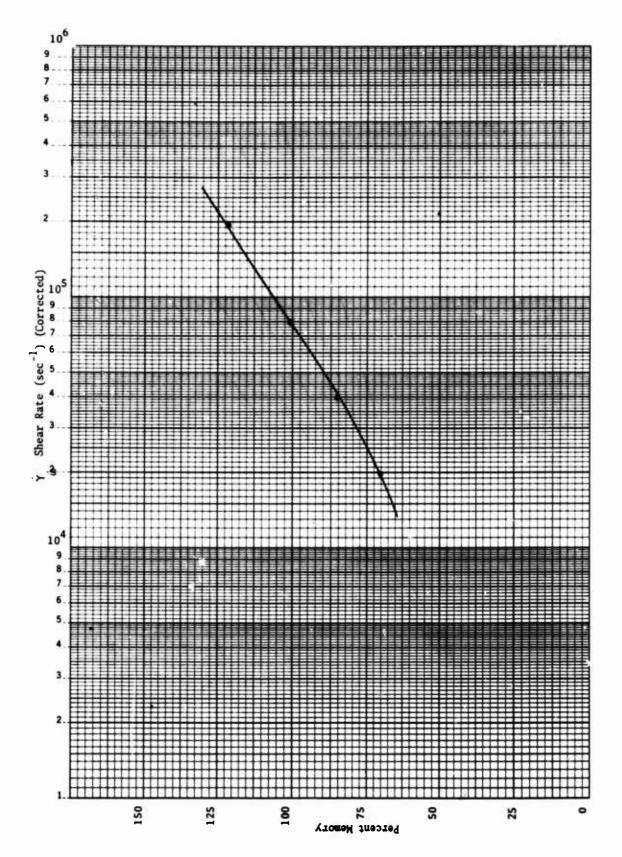


Figure 4. Percent Me ory versus Shear Rate for Napalm B

measurements are required, with an involved analysis procedure to determine the recoverable shear number. In the memory technique, the physical response to the stored energy or the swelling of the material emerging from the capillary is measured. While the technique is more empirical than theoretical, the procedure required for measuring the phenomenon is considerably easier than that for determining the recoverable shear. Either of the methods can be applied to the problem of predicting the behavior of candidate flame fuel formulations on a relative basis. The relationship between recoverable shear and memory is discussed by Bagley and Duffy⁸. It is a complex relationship deeply involved with rheological theory and is beyond the scope of this report.

Reference

^{8.} Bagley, E. B. and Duffy, H. I., "Recoverable Shear, Strain, and the Barnus Effect in Polymer Extrusions," <u>Transactions of the Society of Rheology</u>, 14:4 (1970), p. 545.

SECTION III

EXPERIMENTAL

APPARATUS

The flow data obtained during this study was acquired using a modified Monsanto Research Corporation Model 3501 Capillary Extrusion Rheometer shown in Figure 5. The instrument is designed for measuring the melt flow of thermoplastics. In addition to a capability for determination of the ASTM melt index, provisions are made in the design to vary the temperature from -17.8°C (0°F) to 204.4°C (400°F) and the pressure or stress applied from 0 to 160 psi. The L/r (length to radius) ratio of the capillaries used may be varied over a wide range. Thus, varying the capillaries used and the applied pressure, shear rates to $10^4~{\rm sec}^{-1}$ can be obtained with the basic instrument. Additional modifications to the instrument by the Air Force Armament Laboratory have extended the shear rate range to $10^6~{\rm sec}^{-1}$.

For operation at temperatures of 40°C and below, temperature control is obtained by circulating a cooling fluid through the barrel from an external cooler. The temperature controllers and heaters built into the instrument are used to maintain the desired temperature. Measurements below -6.7°C (20°F) were not attempted because of water condensation inside the barrel.

Pressure is applied to the solution with a ram driven by oil pumped nitrogen in the range of 0 to 160 psi gauge reading. Dual gages are used to improve the precision of the measurements in the lower pressure ranges. The ram designed for the instrument is fabricated of stainless steel with a loose fit to the barrel wall. At higher pressures this loose fit allows some flow-by of material past the ram. To eliminate this flow-by, the ram has been modified by the addition of a Teflon® sleeve on the lower 12 to 15 mm of the ram. (Figure 6 shows the rams used in this study.) This sleeve is slightly tapered with the larger portion at the bottom, thus allowing the Teflon® to flow when under pressure. To compensate for the friction between the Teflon® and the wall, the instrument is calibrated with a Moorhouse Ring Dynamometer with the ram in place to determine the actual force on the solution at each pressure setting at various temperatures.

The capillary is located at the bottom of the barrel and is held in place by a retaining nut shown in Figure 7. The small rod on the nut is used as a reference for measurement of the strand diameter in the determination of elasticity as memory. The capillary length and diameter is selected according to the viscosity of the solution and the shear rates desired. The smaller diameter capillaries made in the Air Force Armament Laboratory machine shop were fabricated from constant bore, type 316 stainless capillary tubing. The capillary tubing was set into the machined capillary

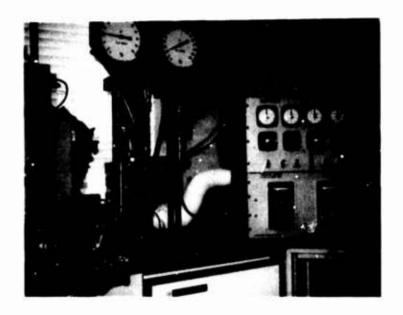


Figure 5. Capillary Extrusion Rheometer



Figure 6. Extrusion Rams used in the Study

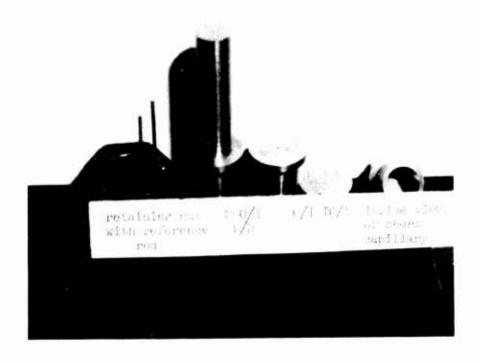


Figure 7. Capillaries used for the Recoverable Shear (S_r) Study

block by freezing the capillary tube and inserting it into the pre-drilled block, which is heated to enlarge the hole for tube insertion. Figure 7 also shows several of the capillaries fabricated by the Air Force Armament Laboratory machine shop. Table I gives a summary of the capillaries used in the study. The dimensions of each capillary used in the study were measured by physical means, using a micrometer to determine the length and an optical comparator to determine the diameter. The viscosities of Brookfield standard solutions were then determined with each capillary to verify these measurements.

The basic rheometer is equipped with four timers which are used to measure the time required to extrude a predetermined volume of material to the nearest 0.01 minute. Using a series of microswitches riding on a cam traveling with the ram, the rheometer in the automatic mode first compresses the fluid, then extrudes four set volumes of material in series from a single filling of the barrel. The time required for each extrusion of 1.245 cm³ (0.0762 in³) is recorded. To obtain higher shear rates or study less viscous materials where the times are very short, a Hewlett-Packard Model 5325B electrical counter (Figure 8), capable of measuring to 0.0001 second, has been wired into the first and last microswitches

TABLE I. CAPILLARY REFERENCE DATA

SOURCE	RATIO	LENGTH	DIAMETER
	L/R	(cm)	(cm)
AFATL	10/1	0.125	0.0250
AFATL	20/1	0.2525	0.0250
AFATL	49.8/1	1.2450	0.0500
MRC	65.7/1	2.5450	0.0775
AFATL	66.9/1	2.5088	0.0750
AFATL	74.9/1	1.8725	0.0500
AFATL	99/1	1.2375	0.0250
AFATL	99.8/1	2.4950	0.0500
AFATL	150/1	1.8750	0.0250
MRC	176/1	1.8608	0.0213
AFATL	200/1	2.5088	0.0250
AFATL	499.8/1	6.2475	0.0250

AFATL - Made by the Air Force Armament Laboratory

MRC - Made by the Monsanto Research Corporation

on the cam. By setting the rheometer to the manual mode of operation, the entire volume of the barrel, $6.751~\rm cm^3$ (0.4119 in³), may be extruded at one time with an accurate measure of the time required; also, the pressure is recorded during the extrusion for each data point.

For the determination of elasticity by the memory technique, the strand of polymer being extruded is photographed with a Singer Super Graphic Camera equipped with a 4 by 5 inch Polaroid® back and modified with a 45.5-cm tube to extend the focal length. This modification gives approximately a 4x magnification on the photograph. The photograph of the strand is then measured with a magnifying comparator, and memory is calculated by the previously mentioned equation.

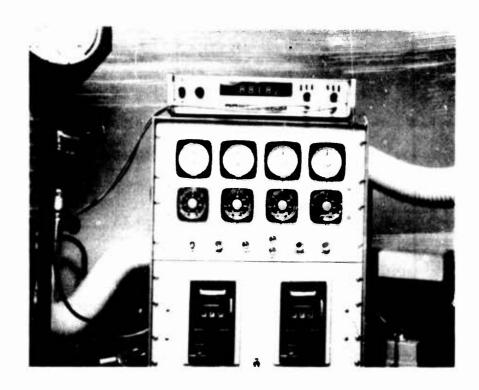


Figure 8 H/P 5325B Electrical Counter

TECHNIQUES

The high volatility and viscous nature of the polymer solutions studied caused problems in loading the rheometer barrel. The procedure used for filling of the barrel must minimize solvent losses and the solution must contain no voids or air bubbles. The technique developed for barrel loading utilized a modified 30 cc disposable syringe fitted with a nominal 0.48 cm x 15.25 cm I. D. piece of Teflon® tubing, as

shown in Figure 9. The sample jar is fitted with a modified lid containing a Swagelok® fitting for tube insertion. The jar is inverted and the syringe filled through this fitting, thus minimizing the solvent loss. The Teflon® tubing is then slipped onto the end of the syringe and the tubing inserted to the bottom of the barrel. The tube is slowly removed as the barrel is filled. When filled, the barrel is sealed with either a rubber stopper or the Teflon® tipped ram. For solutions of low viscosity, the capillary tip is plugged with a round wooden toothpick to prevent solvent losses. The solution is allowed to equilibrate for 5 minutes prior to a run at 23.9°C (75°F) and for 10 minutes prior to a run at 10°C (50°F) or lower.

The orifice is removed and cleaned between each run with a wire reamer and solvents. The barrel is cleaned with a 20-gauge gun barrel cleaner and a soft cloth. After cleaning, the barrel is closed to prevent water condensation at lower temperatures.



Figure 9. Modified Syringe with Tubing

MATERIALS

The Napalm B used in the study was prepared in the laboratory in accordance with the Air Force Armament Laboratory Purchase Description Assignment No. 5, Napalm B, dated 27 April 1966, or procured from Atlas Fabricators, Inc., Torrence, California, as a part of a large purchase of

production material for use in filling experimental weapons. Napalm B is composed of polystyrene (46% \pm 1.0%), gasoline (33% \pm 1.0%) and benzene (21% \pm 1.0%). Regular-grade gasolines were used; most of them contained lead and were obtained from the Eglin Air Force Base motor pool or from local service stations. The polystyrene used was DOW 666, obtained from Dow Chemical Company.

The styrene-butadiene copolymers (SBR) were obtained from the B. F. Goodrich Chemical Company. The two SBRs used in this study were Ameripol® 1513, designated SBR 40 (contains 40 percent styrene) and Ameripol® 1013, designated SBR 43 (contains 43 percent styrene). Solutions of the SBRs were prepared containing a total of 20 percent of one of the copolymers, 43.7 percent gasoline, and 27.3 percent benzene.

The styrene-methylmethacrylate (NAS) used was obtained from the Richardson Company. Solutions were prepared containing 40 percent NAS, 36.9 percent gasoline, and 23.1 percent benzene.

In order to minimize variations in the solutions and eliminate any possible shear degradation of the polymers by mixing, all solutions were prepared by adding the required components to a glass jar, sealing the lid with tape to minimize solvent losses, and slowly rotating the jars on an approximately 66-cm diameter wheel. From two to three days were required to obtain clear solutions with no indications of undissolved material. As an additional check for solvent loss, the prepared samples were weighed before and after mixing.

DATA REDUCTION

The pressure applied for extrusion, the time required for the extrusion, strand diameter measured from the photograph, and the temperature are required for each data point. The data, along with the capillary dimensions and the fixed volume extruded, are used to calculate the shear stress, shear rate, apparent viscosity, and percent memory using a CDC 6600 computer. The computer program is included as Appendix A.

The data generated is presented in graphical form as log-log plots of shear stress versus shear rate and apparent viscosity versus shear rate and semi-log plots of percent memory versus shear rate.

Final curves for the plots of apparent viscosity versus shear rate are determined from a least square analysis of the data points as a part of the computer program.

SECTION IV

RESULTS AND DISCUSSION

CALIBRATION OF EQUIPMENT

In order to verify the uniformity of results from the various capillaries used in the study, a series of runs were carred out using each of the capillaries to measure the viscosity of the same solution. The results of these runs at 23.9°C (75°F) on a sample of Napalm B formulated with regular grade gasoline are shown in Figure 10. All of the data points are well within normal experimental error. This data verifies both the reproducibility of the capillaries and the techniques used to handle the solution and load the rheometer barrel.

Another problem which must be considered is the reproducibility of solution preparation. In order to verify the reproducibility of mixing techniques, three separate solutions of Napalm B formulated with regular grade gasoline were prepared. Figure 11 shows the data obtained from these solutions.

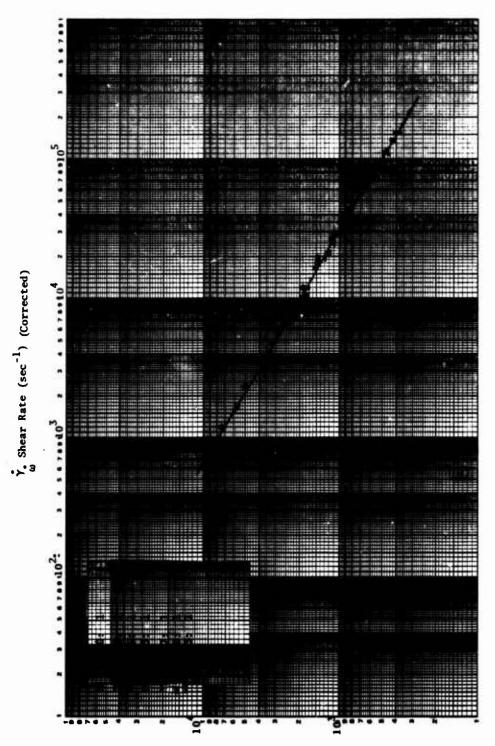
COMPARISON OF RECOVERABLE SHEAR AND MEMORY

The type data obtained from recoverable shear and percent memory determinations can best be illustated by comparing the results of the two methods on the same solution. Figure 12 shows a comparison of the results of the two methods, using Napalm B at 23.9°C as an example. While the numerical values obtained from the two methods are quite different, both values increase as the shear rate is increased. As can be seen from the lines in this figure, the change in memory with increasing shear rate is near linear, while the change in recoverable shear is more drastic and appears to be a logarithmic-type function.

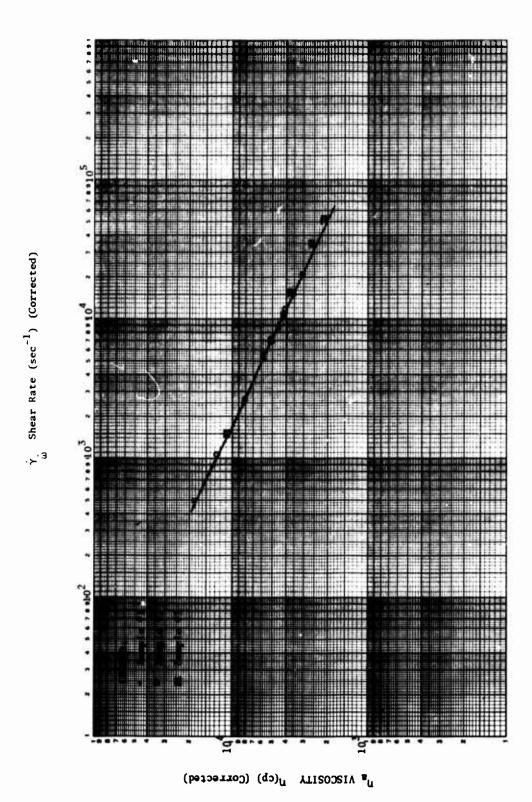
RHEOLOGICAL BEHAVIOR OF NAPALM B

Flow curves (viscosity as a function of shear rate) were prepared for Napalm B at six different temperatures from -6.7°C (20°F) to 60°C (140°F). The results of these experiments, using a production lot of Napalm B, are shown in Figure 13. As has previously been discussed, polystyrene (and thus Napalm B) is a thermoplastic material; that is, its rheological behavior varies as a function of temperature. For the solution studied, the viscosity increases by a factor of about five as the temperature is decreased from 60°C to -6.7°C. The curves obtained are similar to those obtained by Gaskins² at Edgewood Arsenal and Long³, at Monsanto Research Corporation. Small differences in this data and previous data can easily be attributed to actual differences in the solutions, since Long has observed considerable variations from different samples of

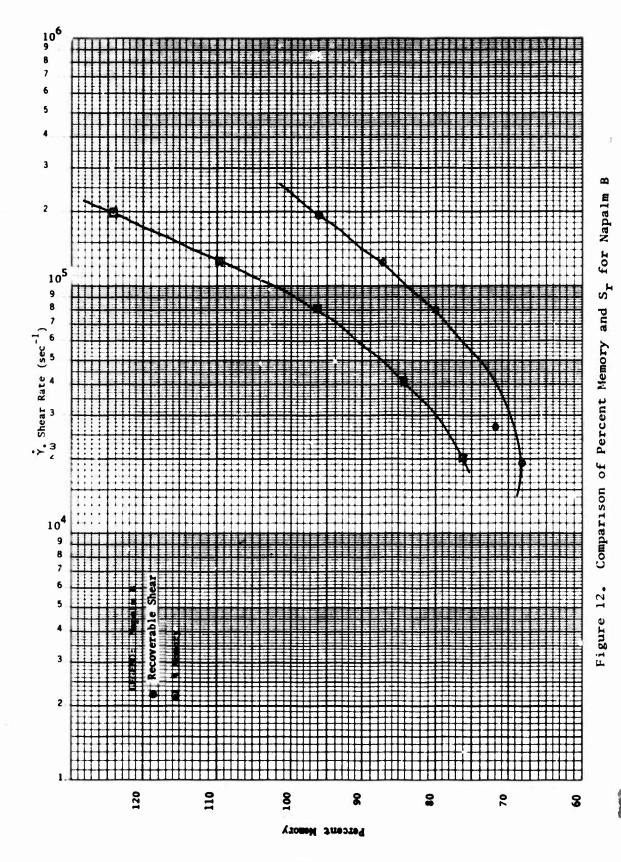


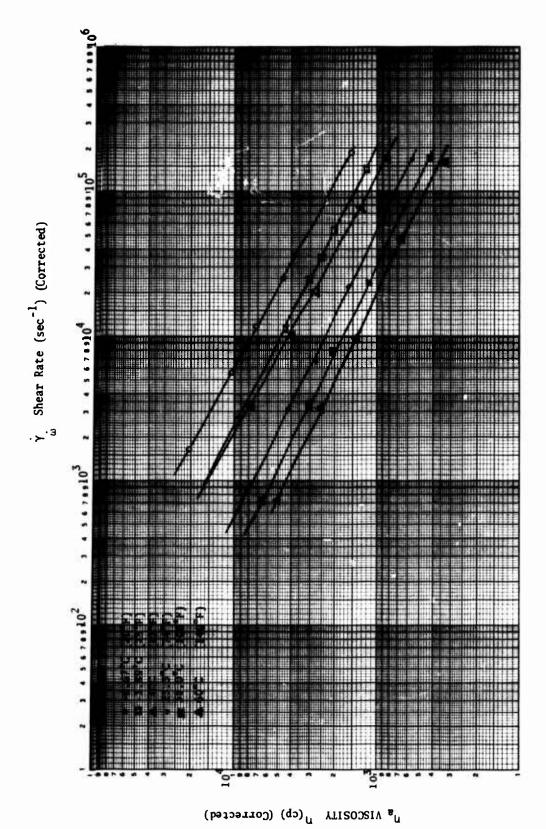


" VISCOSITY cp, (Corrected)



Flow Curve for Napalm B with Chevron Gasoline at OC (32°F)





46 Percent Styrene-Methylmethacrylate at 23.9°C ಡ Flow Curve for

the production lot of Napalm B. These results show that the change in viscosity over the shear rate range of $10^3~{\rm sec}^{-1}$ to $10^5~{\rm sec}^{-1}$ is linear, as compared to the same curvature in the lines reported in previous efforts², ³. Since the primary interest is in the high shear forces during fuel dissemination where a bomb case opens, the lower shear rate range is not of interest to our application and no attempt was made to collect data below $10^2~{\rm sec}^{-1}$.

Figure 14 shows the percent memory of Napalm B as a function of shear rate at -3.89°C and 23.9°C. As was the case for the viscosity, the termoplastic behavior is evident from the considerable difference in the memory at the two temperatures.

The solvent system used to prepare the polystyrene solution has a definite effect on the rheological properties of the solution. To illustrate this, several Napalm B formulations were prepared using different brands of gasoline. The percentages of polystyrene, benzene, and gasoline were constant so the only variable was the brand of the gasoline. Figures 15 and 16 show the effects of various gasolines on the viscosity and memory. Some of the gasolines used did not appear to form a true solution and were not included in the reported studies. As can be seen in the figures, the type of behavior (in the shape and slope of the curves) is the same for the different formulations. However, the absolute values of viscosity and memory are somewhat different for the various gasolines. This behavior emphasizes the importance of controlling experimental variables in a precise laboratory study and illustrates one of the major problems when data from various laboratories is compared.

RHEOLOGICAL BEHAVIOR OF EXPERIMENTAL FORMULATIONS

In previous efforts³, ⁴ several experimental formulations have been evaluated as candidate flame fuels. Three of these formulations are included in this report to illustrate the investigation of different polymer solutions with different properties by this technique.

Figures 17 and 18 show the properties of a 40 percent styrene-methyl-methacrylate (NAS) solution. The elastic properties of this solution are very similar to those of Napalm B, as shown in Figure 18. The viscosity is slightly less dependent on shear rate than that of Napalm B; i.e., the viscosity versus shear rate curve is more horizontal. In addition, the temperature dependence is even more pronounced than for Napalm B. Based on this information, it would be predicted that NAS would be equal to or slightly better than Napalm B at 75°F; however, the increased viscosity at 25°F indicates that the performance would be inferior at low temperatures.

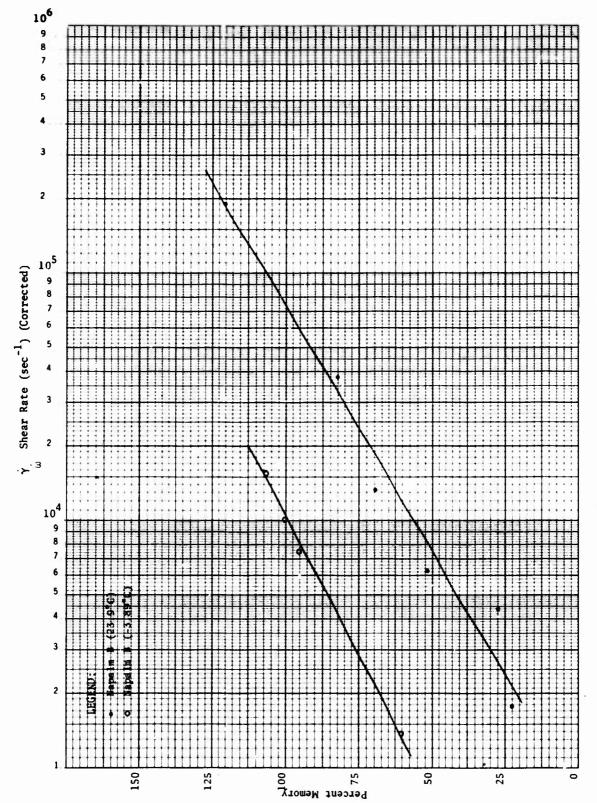
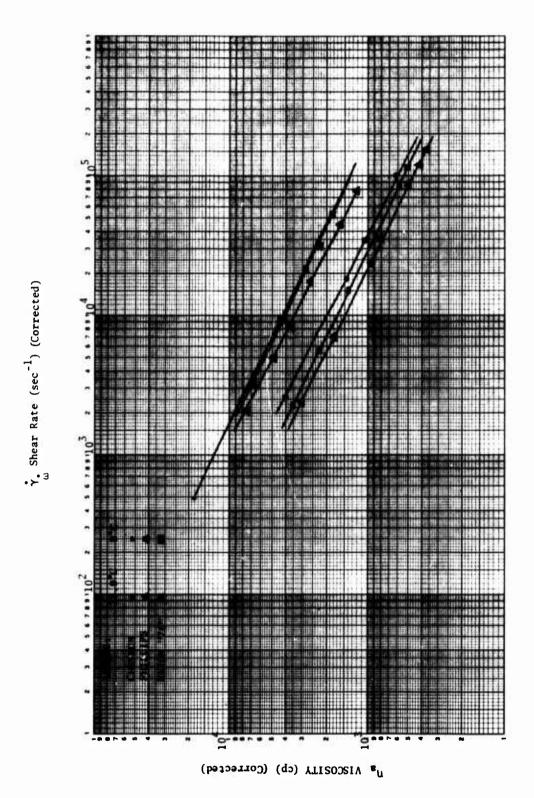


Figure 14. Percent Memory of Napalm B (Dow Mixed)



Flow Curve of Napalm B Formulated with Various Gasolines Figure 15.

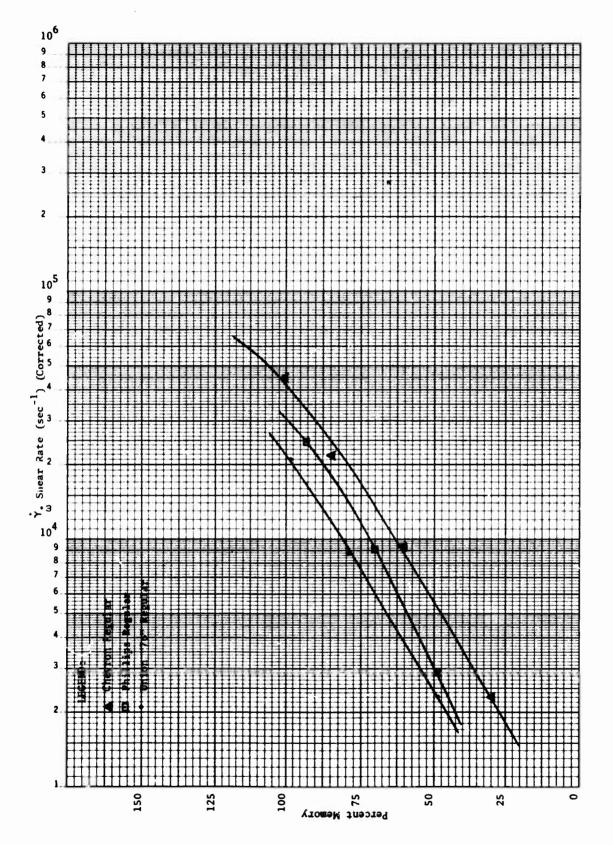
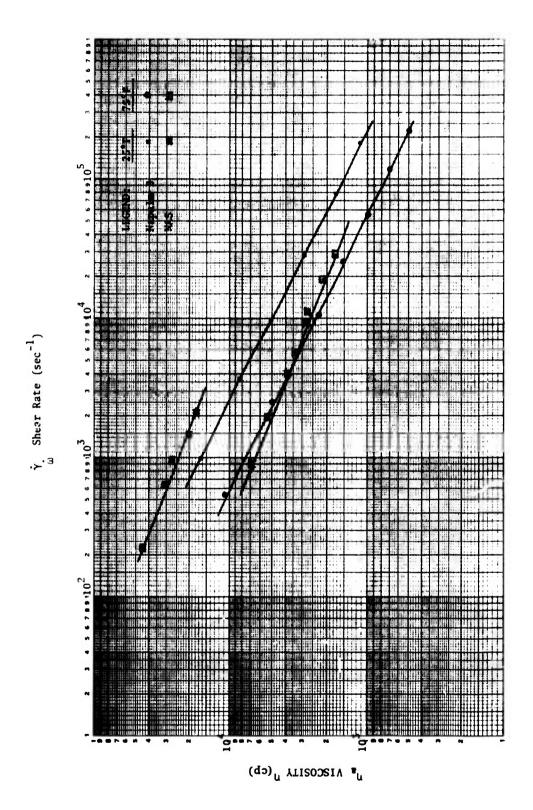
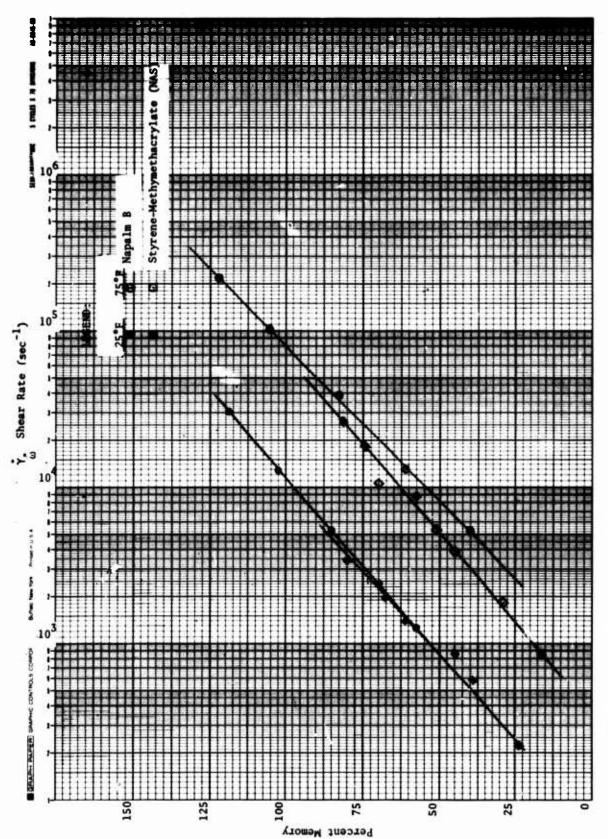


Figure 16. Percent Memory of Various Gasolines and Polystyrene Formulations



Flow Curve of Napalm B and Styrene-Methylmethacrylate at 25°F and 75°F Figure 17.



a 40 Percent Formulation Comparison of Percent Memory for Napalm B and of Styrene-Methylmethacrylate (NAS) Figure 18.

The most promising candidate materials investigated to date have been the styrene-butadiene (SBR) copolymers. The viscoelastic properties of these rubber-like materials are considerably less temperature dependent than the properties of a thermoplastic material like polystyrene. In addition, the viscosity and elasticity can be varied by changing the percentage of styrene in the copolymer and by altering the molecular weight distribution.

The properties of a 29 percent solution of SBR 40 (containing 40 percent styrene) are shown in Figures 19 and 20. Although the viscosity is lower than that of Napalm B, it is significantly less temperature dependent. The elastic properties are almost independent of the shear rate, contrasting sharply to the steep shear rate dependence of Napalm B.

Figures 21 and 22 show the properties of a 29 percent solution of SBR 43 (containing 43 percent styrene). The rheological properties are very similar to those observed for SBR 40, except the absolute value of percent memory is higher; that is, the solution has a greater elastic strength.

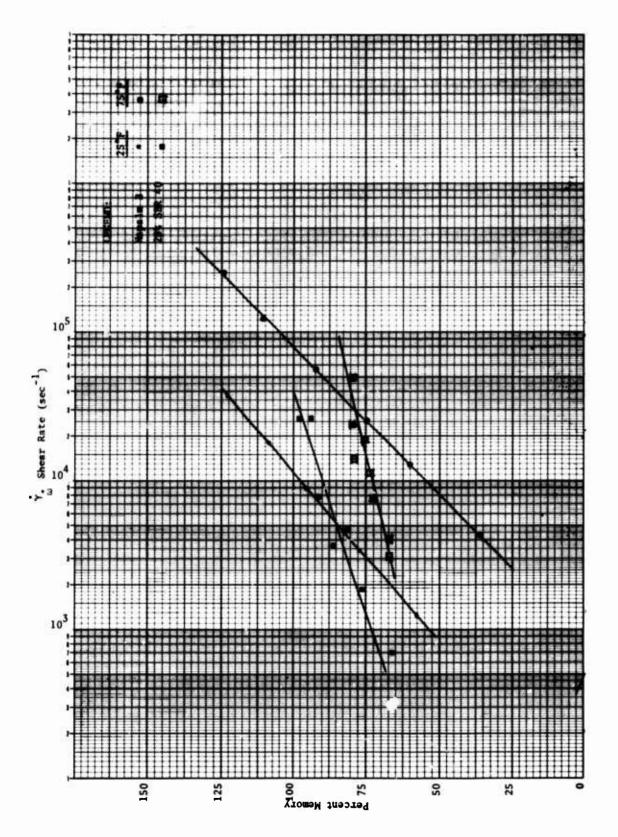
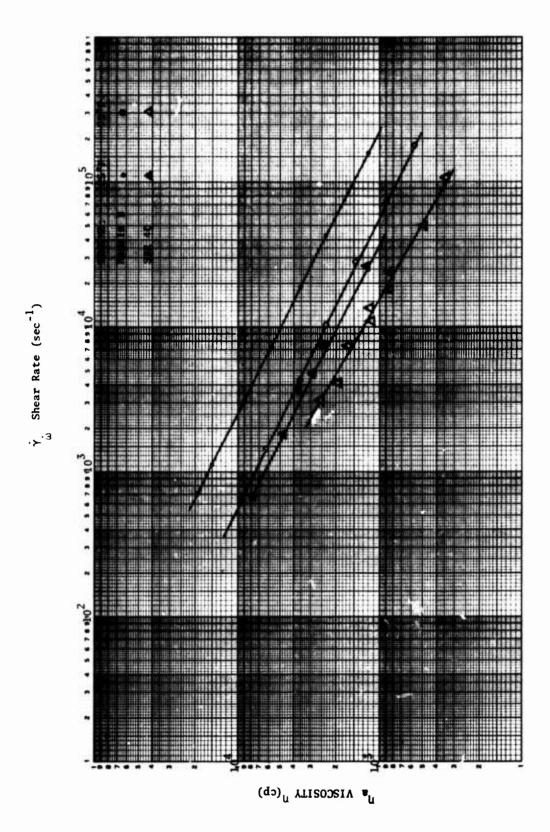


Figure 19. Comparison of Percent Memory for Napalm B and 29 Percent SBR 40 Solution



Comparison of Viscosity versus Shear Rate of 29 Percent SBR

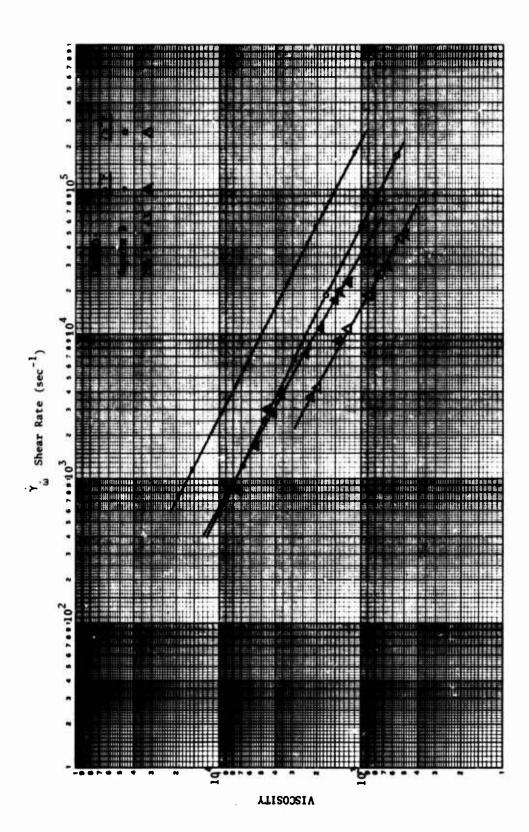
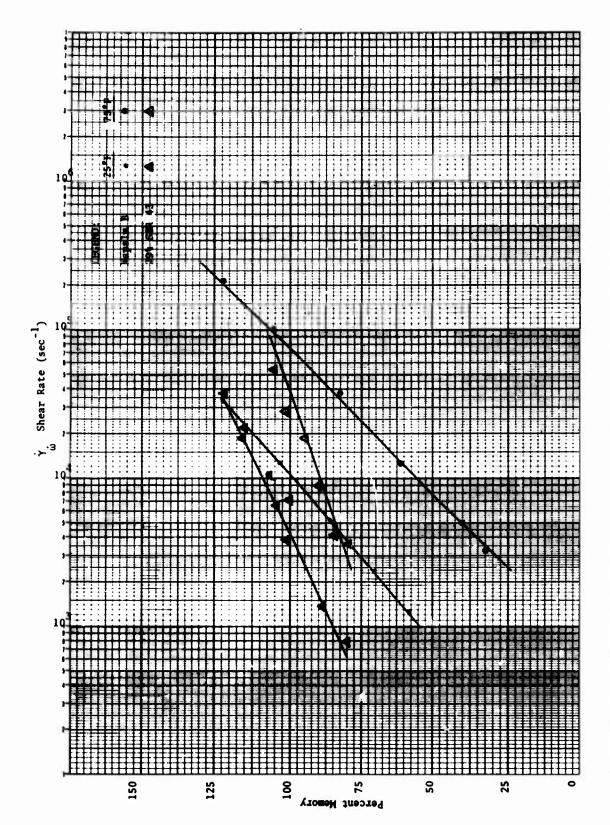


Figure 21. Comparison of Viscosity for Napalm B and a 29 Percent SBR 43 Formulation



29 Percent SBR 43 Formulation Comparison of Percent Memory for Napalm B and

SECTION V

SUMMARY AND CONCLUSIONS

The instrumentation and techniques developed during this study represent a significant advancement in the Air Force capability for development of improved flame agents for use in firebombs. The study has shown that a capillary extrusive rheometer can be utilized to measure the rheological properties of polymer solutions with viscosities in the range of 10^2 to 10^5 centipoise at shear rates from 10^2 to 10^6 sec⁻¹, as a function of temperature from 25° F to 100° F.

The rheological behavior of a given polymer solution can be utilized to predict the dynamic behavior of the material during dissemination from a firebomb. Thus, this laboratory-scale technique can be used to screen candidate firebomb fuels.

While either the die swell or recoverable shear methods may be used to study the elastic properties of a solution, the die swell method has been shown to be a much faster and simpler method of obtaining the desired information.

The solvent system used in preparation of a solution has been shown to have an effect on the values of viscosity and elasticity of a solution but not on the shapes of the curves. A series of curves obtained for solutions prepared with different brands of gasoline are parallel but offset in the value of viscosity or elasticity. These results emphasize the importance of using a standardized gasoline or gasoline simulant when data from different techniques or different laboratories are compared.

The rheological properties of solutions of the styrene-butadiene copolymers (SBR) have been shown to be less dependent on temperature or shear rate than thermoplastics, such as polystyrene.

More data are required to determine the extent of shear degradation with a given viscoelastic property. However, the test used did not attempt to measure all the controlling parameters encountered in dynamic air gun and sled track tests. It should be recognized that the test used in this study was designed for laboratory purposes and does not necessarily predict the field behavior of the agent when exposed to various means of dissemination.

APPENDIX A

CAPILLARY EXTRUSION RHEOLOGY DATA REDUCTION PROGRAM 1939

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100	CALL TITLE(ITEMP, CAP, CLEW, CDIA, ID, IM, IV, SAMPLE)	P1939	111
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	CALL	P1939	104
165	C COMPUTE CORRECTION FACTOR	P1939	165
	CORF # (8+3.)/4.	P1939	186
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	C COMPUTE CORRECTED RATE OF SHEAR AT WALL	P1939	108
	00 45 I = 1,00F	P1939	109
110	SRC(I) = CORFFC(I)	P1939	110
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	4N CONTINUE	P1939	112
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	C COMPUTE APPARENT CORRECTED VISCOSITY AT WALL (VISN)-CENTIPOISE UNITS	P1939	114
115		P1939	115
	50 VISN(I) H T(I) /SRC(I) +100.	P1939	116
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	C PLOT - VINN WA REC ON 106-106 PAPER	P1939	118
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	C Y-AXIS- VISA X-AXIS - SRC	P1939	121
	CALL SETMIV(100.54.54.100)	P1939	122
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125	IF(SSMIN.LT.1000.) XLIM = 150.	P1939	125
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94.	CALL TITLE (ITEMP.CAP.CLEN.COIA.IO.IM.IY.SAMPLE)	P1939	130
	C VERTICAL AND HORIZCHTAL LABELS	P1939	131
	ALL	P1939	132
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	CALL RITEZV(10,658,1024,90,1,1,-1,1HA,ML)	P1939	134

135	137	138	139	140	141	142	143	166	145	146	147	140	149	150	151	152	153	154	155	156	151	158	159	160	161	162	163	164	165	166	167	100	27	171	172	173	174	175	176	177	178	179	168	161	182	183	101
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	PITE2V(10,603,1024,90,1,1,-1,146,NL)	61614	187
	RITEZV(10,584,1824,98,1,1,-1,14R,NL)	6£6 7 d	168
	FITE2V(18,565+1024,90,1,1,-1,14C,NL)	62614	189
191	1.11.1HE,NL)	P1939	190
	RITECVI10,527,1024,90,1,1,-1,14N,NL)	61614	151
	RITE2V(10,508+1024,98,1.11.1HT.NL)	P1939	192
	RITE2V(10.470.1924.90.1.11.14M.NL)	P1939	193
	1,-1,1HE,NL)	P1939	18
195	RITE2V(10,432,1024,90,1,1,-1,1HM,NL)	P1939	195
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230	GO TO 1EOF, (150,1000)	P193900	•
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	C.EQ.1) CALL TITLE(ITEMP,CAP,CLEN,COIA,ID,IM,IY,SAMPLE)	P1939	232
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	END	P1939	7

SUBI	SUBROUTINE LOGFIT	LOGE	T 76/76 CPT=1	FTN 4.2+ REL	12/02/15	19.29.32
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			SUBROUTINE LOGFIT(X,Y)		626Td	424
		L			P1939	425
			SUBBOUTINE FOR LOG FIT		P1939	426
	_				P1939	427
v			DIMENSION X(20) -Y(20)		P1939	428
•					P1939	624
			O'- XETY		P1939	430
					P1939	431
					P1939	432
0					P1939	433
			DO 5 K=1 -NOF		P1939	424
			TF(X(K) = E0.000)XX= 0.0001		6163 6	435
			TF(Y(K)_E0_0_0) Y(K) = 0.0001		P1939	436
					P1939	437
¥			XX = ALOGIO (X(K))		P1919	438
`			XX+XXX = XIIX		P1939	439
			SUMX2 = SUMX2+XX+XX	74	P1939	944
			SUMLY = SUMLY+ALY		P1939	441
			SMXLY = SMXLY+XX #ALY		P1919	244
6		S	5 CONTINUE		P1939	644
:		•	10X # ZU		P1939	455
			DE = (FN+S')4X2) - (SUMX+SUMX)		P1939	445
			TE(0) - FO. 0. 0) GO TO 10		P1939	944
			AL = ((SUMLY*SUMX)) /OL		P1919	255
ď			RI # ((FN*SAXLY) - (SUMX#SUMLY)) /OL		6161d	844
					P1939	674
					6161d	450
			YCT = A+X(1)++B		P1939	451
			Station Nation		6161d	259
5			CALL LINEV(NXV(X(1)), NYV(YST), NXV(X(NOF)), NYV(YLT))	IYLTII	b161d	453
			CALL LINEV(NXV(X(1)), NYV (YST), NXV (X (NOF)), NYV ((YLT))	P£939	454
			RETURN		P1939	455
		-	PRINT 161		P1939	456
		101	FORMAT (140, *COFFFICIENT DET. IS 0. NO SOLUTION*)	•	P1939	457
5		 	URN		P1933	458
			ENO		6161d	459

	SURROUTINE FIT	FIT	74/74 CPT=1 F	FTN 4.2+ REL	12/02/15	03.29.25
			SUBROUTIME FIT (x, Y)		P1939	399
	C	,.			P1939	100
	. 0	C SU	SUBROUTINE FOR LEAST SQUARES FIT FOR LINEAR PLOTS		P1939	401
					P1939	402
5			COMMON NOF		P1939	663
•			COMMON /FIT1/ E		P1939	+0+
			DIMENSION X(25) . Y(20)		P1939	405
			SUMXY = B.		P1939	436
			SUMY # 0.		6167d	200
10			SURX II D.		P1939	408
			SUMX2 = 0.		P1939	603
			DO 10 I=1.NOF		P1939	410
			SUMX = SUMX+X(I)		P1939	411
			SUMX2 = SUMX2+X(I)+X(I)		P1939	412
15	,.		SUMY = SUMY+Y(I)		P1939	413
•		10	SUMXY = X(I) +Y(I) +SUMXY		P1939	414
		1	D = NOF#SUMX2- (SUMX#SUMX)		P1939	415
			B = ((NOF*SUNXY)-(SUNX*SUNY))/0		P1939	416
			A = ((SUMX2*SUMY)-(SUMX*SUMXY))/D		6161d	417
20			YST = A+8*X(1)		P1939	418
			YEND # A+8*X(NCF)		P1939	419
			CALL LINEV(HXV (X(1)), NYV (YST), KXV (X(NOF)), NYV (YEND))	(CON:	P1939	420
			CALL LINEY(NXY (X(1)), NYY (YST), NXV (X (NOF)), NYV (YEND))	(CON:	P1939	421
			RETURN		P1939	452
5.			END		P1919	623

SUBROUTINE RECOVE	RECOVE	76/76 CPT=1	FTN 4.2+ REL	12/32/75	99.29.20.
	Š	SUBSOUTINE RECEVE		P1939	293
				P1939	7ó2
	ى د			P1939	295
	C THIS	ROUTINE	2 250-1 FOR	P1939	962
2	C RECOV	COVERABLE SHRESS NUMBER CALUCATIONS.		6161d	297
	ပ			P1939	296
	īO	DIMENSION TLOG(20), SRC(20), TLOG1(20), SRC1(29), DUM(20	DUM (20)	61614	662
	CO	COMMON NNOF, XMBX, XMIN, YMBX, YMIN		P1933	360
	X	EXTERNAL TAGLIV		6161d	361
1,	00	0 5 I=1,20		P1939	362
	5 00	DUM(I) = 0.		6161d	303
	RE	1 0 1		P1939	354
		XIS TLOG Y-AXIS SRC		P1939	305
	C FIRST C	ST CAPILLARY		P1939	306
15	RE	READ(1) NOF1, (TLOG(I), I=1,NOF1), XMIN1, XMAX1, CAP1, CLENI	#P1.CLEN1	P1939	307
	RE	READ(1) (SRC(I) , I=1,NOF1)		61616	308
	C SECOND			5£61a	369
		0	CAP2, CLEN2	P1939	310
	A.	EAD(1) (SPC1(I), I=1,NOF2)		91919	311
23	IF	IF(EOF(1), NE. 0.0) CALL EXIT		P193900	ī.
	C MAX AND	AND MIN K FOR PLOTS		61616	312
		v		P1939	313
	ž ×	10		P1939	314
	CCONVER	۰.		6161d	315
25		0 I=1,NOF1		6161d	316
	10 580	F		P1939	317
		2		P1939	318
	15 SR			P1939	319
		NNOF & NOFL		P1919	320
3.0	CA			P1913	321
	××	z		P1939	322
	XX	H		6£61d	323
	Ž	MNOF = MOF2		P1939	326
	CAL	CALL MAXMINIDUP, SRC1)		P1919	325
35	C MAX A!	AND MIN Y FOR PLOTS		P1919	326
	N A			P1939	327
	¥ >	YHAX = AMEX1(XXMEX, YHAX)		P1939	328
	W.X	SAIR - NEELS		P1939	329
	X	14		P1939	330
9	11	### HI		6161d	331
	וו	M		P1939	332
	IF	IF ((XMAX-XMIN), GT.2.) LL = 2		P1939	333
	¥I	IF ((YMAX-YMIN) GT.2.) LLL = 2		P1939	334
	X	= XMAX+.10060		P1930	335

4.5	YMAX = YMAK+.20000	P1 939	900
		61614	337
	MIDA 40 100 INTXA	P1939	338
	WI 12 (89 1)	61616	339
	KITE (# 5) (SKC(I) · I & I & I & MO	P1939	200
26	WRITE(8,3) CAP2,(TLOG1(I),1=1,NOF2)	P1939	341
	RITE(6,2) (SRC1(I),I=1,NOF2)	P1939	342
	1 FORMAT(1M0.9X,*RECOVERABLE SHEAR NUMBERS*/10X,*CAPILLARY *.A7/	P1939	343
	1 16xee x a e-0xe (10f0.t))	P1939	440
	2 FORMAT(10x, *Y = *, 3x, (10F8.4))	P1939	345
53	3 FORMAT(10x, +CAFILLARY +, A7/10x, + x = +, 3x, (10F8, 4))	P1939	346
	W(100.54.54.100)	P1939	347
	(0.0)	P1939	946
	CALL GRIDLACS.xmix.xmax.ymix.ymax	91919	545
	V(2,2)	P1939	350
90	1) /	P1939	351
		P1939	352
٠		P1939	353
		P1939	354
	CALL RITE2V(10.050.1624.90.1.11.1MG.ML)	P1939	355
65	V(13.831.1324.90.1.1	P1939	356
	V (10	P1939	357
	1 (10 , 793, 1324, 90, 1, 1, 1, 1	P1939	358
	V(10,774,1024, 30	Pigs	359
	2V(10,736,1924,90,1,1,-1	P1939	360
7.0	V (13,717,1024,98	P1939	361
	V(10,698,1024,90,1	P1939	362
	* (10,660,1024,90,1,1,-1,1H.,	P1919	363
	V (10,622,1924,90,	P1939	364
	V(10,603,1024,90,	P1919	365
75	V(10,594,1024,90.	P1939	366
	24 (19	P1939	367
	V(10.546.1924.90.1.1	1010	36.8
	217 7	P1939	592
	01) A	P1979	376
60	V (10	P1939	371
	17 (1	P1939	372
	CALL RITE2V(10,432,1024,90,1,1,-1,1145,NL)	P1939	373
	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	P1939	374
	V(10,394,1624,90	P1939	375
# 2	V(10,375,1024,90	biold	376
	V(10,356,1024,90	61614	377
	V(10,318,1024,90,1	P1939	378
	:V(10,299,1024,98,1	P1939	6/5
	V (10,281,1024,90,1,1	61614	
6	1V(10,262,1024,90,1,1,-1,1HE,NL)	61614	125
	4,968,1024,90,1,22,-1,22HCAPILLARY UIA	61614	
	· A (+ + + · 30 0 ·	2 5	
	. CLENN) CLEN1	5	# L
	ENCODE (6,4,CLEN1) CLEN2	61434	606

```
4 FORMAT(F6.4)
CALL RITEZV(587,968,1024,90,1,6,1,CLENN,NL)
CALL RITEZV(691,968,1024,90,1,6,1,CLENN1,NL)
CALL APLOTV(NOF1,TL06,SRC,1,1,1,1M*,IERR)
CALL APLOTV(NOF2,TL061,SRC,1,1,1,1,1,38,IERR)
CALL APLOTV(NOF2,TL061,SRC,1,1,1,1,38,IERR)
NNOF = NOF1
CALL FIT(TL06,SRC)
NNOF = NOF2
CALL FIT(TL06,SRC)
RETURN
ENOF
   .
                                      100
                                                                           105
   95
```

SUBROUTINE TITLE	NE	TITLE	76/76	CPT=1	ie.	FTN 4.2+ R	REL	12/03/75	09.29.15.
		SUBF	ROUTINE T	ITLECITE	EMP.CAP.CLEN.CDIA.ID.IM.IY.S	AMPLEI		P: 939	263
		OINE	ENSTON SA	APLE(6)	DIMENSION SAMPLE(6)			P1919	264
	U	THO	NES OF HEADERS ON PLOTS	ADERS ON	N PLOTS			P1939	592
		CAL	L PITEZV	54.1000	0, 1724, 90,1,6,-1,6HSAMPLE, NL	_		6£61d	992
·		CALL	L RITEZV	159.1000	L RITEZV (15%, 1000, 1924, 90, 1, 53, 1, SAMPLE, NL)			6161d	267
•		IFIC	CLEN.LT.0.) 60 TO 25	.) GO TO	0 25			P1939	268
		CALL	L RITEZV	54.980.	.1024.90.1.91.9HCAPILLARY.	<u>ال</u>		P1939	569
		CALL	L PITEZV	194.390.	L PITE2V(154,390,1024,90,1,7,1,CAP ,NL)			6161d	276
		CALL	L RITEZV	304.980.	.1024.90.1.31.3HL =.NL)			6161d	271
2		ENC	00E (6.21.	CLENN) CL	Nul			P1919	272
		21 FORP	MAT (F6.4)					P1919	273
		2	L RITEZV	356.980.	.1024.90.1.5.1.CLENN.NL)			P1919	274
		CALL	L RITEZV	.66.990	L RITEZY(460.980.1824.90.1.31.3HD =.NL)			6£67d	275
		PNC	00E (6 . 21 .	COIAA)CD	DIA			blöld	276
<u>v</u>		C 4 L L	L RITEZV(512.980.	L RITEZV(512,980,1024,90,1,6,1,CDIAA,NL)			6161d	277
		30 CALL	L RITEZV	51E.980.	L RITEZV (616,980,1024,90,1,6,-1,6HTFHP =,NL)			P1979	278
		N	ODE (3.22.1 EMP1) ITEMP	I (EMB.)				P1939	279
		22 FOR	FORMAT (13)					P1939	280
		CAL	L RITEZVI	727.980.	.1024.90.1.3.1.ITEMP1.NL)			P1 9 39	281
-		CALL	RITEZV	7.55.980.	.1024.90.1.11.1HF.NL)			P1030	282
		CALL	L KITEZVI	796.367	L KITEZV(/98,988,1024,98,1,2,1,10,NL)			P1939	243
		CALI	L RITEZV	637,980.	.1024,90,1,3,1,IM,NL)			P1919	286
		CALI	L PITEZV	889.988	.1024.90.1.2.1.IY.NL)			P1939	285
		ארבי	CPN					P1939	286
٠,			L PITEZV	54.990.1	L RITEZV(54,980,1024,30,1,25,-1,25HPECOVERABLE SHEAR	LE SHEAR	NUMBERS.	6261d	287
.								£161d	298
		D'A'B	ENCODE (5.21.COIAA) CDIA	COIAA)CD	DIA			£161d	289
		CALI	L PITE2V(340,960,	.1024,90,1,6,1,CDIAA,NL)			c£51a	352
		0.5	TO 36		60 TO 36			tiold	291
C 8		END						61010	262

	SUBROUTINE MAXMIN	MAXMIN	74/74	CPT=1	FTN 4.2+ REL	12/62/75	09.29.10.
		a di	APOLITING MA	(A.X)		P1939	238
		֓֞֝֞֜֜֝֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓	MENSTON X 12	CONTRACTOR ACTION OF THE PROPERTY OF THE PROPE		P1939	239
			THUN NOTE AND	ZISATXSAATISATXS		P1939	240
		A X X	TAN MEN CA	TEC FOR ELLTS		P1939	241
u			A B B B B B B B B B B B B B B B B			6161d	242
r		**	TN - DOGO.			P1939	243
		× >	1666 - XT			P1539	244
			Tel = 0000.			P1939	545
			40 T=1 NOF			6161d	546
		9	1 2	AAY) XMAX = X(T)		P1919	247
4		4 6	7×	W N		P1939	248
			CATA AD CATAN			P1939	549
			CALMA TI CELAN			P1939	250
			MTTELLE			P1933	251
•			MIN ON WAN	MITHITS FOR PLOTS		P1939	252
13		1000	D'AXWX - X			P1939	253
			- X - X - X			P1979	254
			A - VMAYE. 2			P1919	255
						P1939	256
			W (2) (2)	v		P1933	257
2		7 7		2		P1939	258
		· ·	STATE OF THE STATE	ين		P1933	652
		***				P1939	26.3
			MOILE			6101d	261
		֓֞֝֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓֓				P1939	262

SYMBOLS

L/D	length to diameter of capillary
ASTM	American Society for Testing and Materials
sec ⁻¹	reciprocal second
F	force in pounds or dynes
Na	absolute viscosity, poise
R	barrel radius, cm
r	capillary radius, cm
L	capillary length, cm
Q	extrusion rate, cm ³ /sec
Ϋ́ ω	shear rate, sec ⁻¹ (corrected)
ср	centipoise
b	slope of flow curve
P	pressure difference (dynes/cm ²)
T	shear stress (dynes/cm ²)
$s_{f r}$	recoverable shear number
D	diameter strand or capillary diameter

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